

MEMORANDUM

TO: Mr. Joseph Lysonski
Anderson, Mulholland and Associates

DATE: February 19, 2012

FROM: R. Infante 

FILE: JA98691

RE: Data Validation
BMSMC, Building 5 Area, PR
SM04.00.06

Accutest Job Numbers: JA98691

SUMMARY

Full validation was performed on the data for several soil gas samples analyzed for selected volatile organic compounds (VOC's) by method Compendium Method TO-15. Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999 and Compendium Method TO-3. METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR USING CRYOGENIC PRECONCENTRATION TECHNIQUES AND GAS CHROMATOGRAPHY WITH FLAME IONIZATION AND ELECTRON CAPTURE DETECTION, Revision 1.1, April, 1984. The samples were collected at the BMSMC, Building 5 site, Humacao, PR on February 2, 2012 and submitted to Accutest Laboratories of Dayton, New Jersey that analyzed and reported the results under delivery group (SDG) JA98691.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Compendium Method TO-15. Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999 and Compendium Method TO-3. METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR USING CRYOGENIC PRECONCENTRATION TECHNIQUES AND GAS CHROMATOGRAPHY WITH FLAME IONIZATION AND ELECTRON CAPTURE DETECTION, Revision 1.1, April, 1984. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

SAMPLES

The samples included in the review are listed below

FIELD SAMPLE ID	LABORATORY ID	ANALYSIS
I-7A	JA98691-1	VOCs; Methane
A-1 R3A	JA98691-2	VOCs; Methane

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- Agreement of analysis conducted with chain of custody (COC) form
- Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- Canister cleaning certification criteria
- Surrogate spike recovery
- Internal standard performance and retention times
- Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form.

Holding Times and Sample Preservation

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

GC/MS Tunes

The frequency and abundance of bromofluorobenzene (BFB) tunes were within the QC acceptance criteria. All samples were analyzed within the tuning criteria associated with the method.

Initial and Continuing Calibrations

VOCs

The percent relative standard deviations (%RSDs) and response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Correlation coefficients (r^2) of target analytes were within the QC acceptance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard.

Methane

The percent relative standard deviations (%RSDs) and response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Correlation coefficients (r^2) of target analytes were within the QC acceptance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks for VOCs.

No field/equipment/trip blanks were analyzed associated with this data set.

Surrogate Spike Recovery

The surrogate recoveries were within the laboratory QC acceptance limits in all samples analyzed except for the followings:

Method TO-15

SAMPLE ID	SURROGATE COMPOUND	ACTION
	4-BFB	
JA98691-1	44.0%	No action taken; the
JA98691-2	47.0%	method does not
		have a surrogate
		recovery criteria.

Internal Standard Performance

VOCs

Samples were spiked with the method specified internal standard. Internal standard are performance and retention times met the QC acceptance criteria in all sample analyses and calibration standards.

Field Duplicate Results

VOCs

Laboratory/field duplicate were analyzed as part of this data set F89892-1/-DUP. Results were within laboratory/recommended control limits except for the following:

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
Toluene	0.80	0.65 I	0.49 J	40	No action taken concentration < 5 x SQL

Methane

Laboratory/field duplicate were analyzed as part of this data set JA98412-1/1-DUP. Results were within laboratory/recommended control limits.

LCS/LCSD Results

VOCs

The laboratory analyzed one LCS (blank spike) associated with each matrix from this data set. The % recoveries of all spiked analytes were within the laboratory QC acceptance limits.

Methane

The laboratory analyzed one LCS (blank spike) associated with each matrix from this data set. The % recoveries of all spiked analytes were within the laboratory QC acceptance limits.

Quantitation Limits and Sample Results

Concentration over calibration range in sample JA98691-1, laboratory qualified result as E; sample result qualified as estimated (J).

Dilutions were required with this data set.


Method TO-15

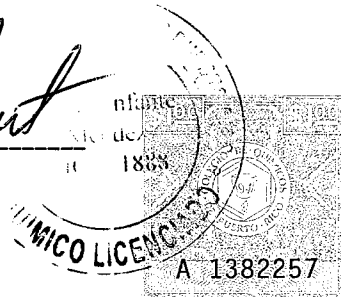
Sample	Dilution Factor	Reason for Dilution
JA98691-1	220 X; 11,000 X	Several VOCs outside calibration range
JA98691-2	240 X; 12,000 X	Several VOCs outside calibration range

Calculations were spot checked.

Certification

The following samples JA98691-1 and JA98691-2 were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. Some of the results were qualified. The results are valid.


Rafael Infante
Chemist License 1888



Report of Analysis

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Client Sample ID:	I-7A	Date Sampled:	02/02/12
Lab Sample ID:	JA98691-1	Date Received:	02/04/12
Matrix:	AIR - Soil Vapor Comp.	Summa ID:	A724,A729,A592
Method:	TO-15	Percent Solids:	n/a
Project:	BMSMC, Building 5 Area, PR		

Run #1	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	3W26094.D	220	02/07/12	YXC	n/a	n/a	V3W1029
Run #2	3W26109.D	11000	02/08/12	YXC	n/a	n/a	V3W1030

Run #1	Initial Volume
Run #1	40.0 ml
Run #2	200 ml

Purgeable Aromatics

CAS No.	MW	Compound	Result	RL	MDL	Units	Q	Result	RL	Units
71-43-2	78.11	Benzene	312	440	100	ppbv	J	997	1400	ug/m3
108-88-3	92.14	Toluene	463000 ^a	4400	880	ppbv		1740000 ^a	17000	ug/m3
100-41-4	106.2	Ethylbenzene	448000 ^a	4400	670	ppbv		1950000 ^a	19000	ug/m3
1330-20-7	106.2	Xylenes (total)	1090000 ^a	4400	680	ppbv		4740000 ^a	19000	ug/m3
	106.2	m,p-Xylene	1070000 ^a	4400	680	ppbv		4650000 ^a	19000	ug/m3
95-47-6	106.2	o-Xylene	21100	440	68	ppbv		91600	1900	ug/m3
67-64-1	58.08	Acetone	50400	440	80	ppbv		120000	1000	ug/m3
67-63-0	60.1	Isopropyl Alcohol	116000 ^a	4400	1300	ppbv		285000 ^a	11000	ug/m3
108-10-1	100.2	Methyl Isobutyl Ketone	815000 ^a	4400	800	ppbv		3340000 ^a	18000	ug/m3

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	4-Bromofluorobenzene	44% ^b	95%	65-128%

(a) Result is from Run# 2

(b) Outside control limits due to matrix interference.



ND = Not detected MDL - Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

Accutest LabLink@657856 13:40 15-Feb-2012

Report of Analysis

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Client Sample ID:	I-7A	Date Sampled:	02/02/12
Lab Sample ID:	JA98691-1	Date Received:	02/04/12
Matrix:	AIR - Soil Vapor Comp. Summa ID: A724	Percent Solids:	n/a
Method:	EPA TO-3		
Project:	BMSMC, Building 5 Area, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	QR93714.D	1	02/07/12	TCH	n/a	n/a	GQR4341
Run #2							

Run #	Initial Volume
Run #1	0.50 ml
Run #2	

CAS No.	MW	Compound	Result	RL	MDL	Units	Q	Result	RL	Units
74-82-8	16	Methane	2350 J	5.0	0.74	ppmv	E	1540	3.3	mg/m3

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	4-Bromofluorobenzene	98%		81-115%
460-00-4	4-Bromofluorobenzene	102%		81-115%



ND = Not detected MDL - Method Detection Limit
RL = Reporting Limit
E = Indicates value exceeds calibration range

J = Indicates an estimated value
B = Indicates analyte found in associated method blank
N = Indicates presumptive evidence of a compound

Report of Analysis

Page 1 of 1

Client Sample ID:	A-1R3A	Date Sampled:	02/02/12
Lab Sample ID:	JA98691-2	Date Received:	02/04/12
Matrix:	AIR - Soil Vapor Comp.	Summa ID:	A525,A535,A538,A543
Method:	TO-15	Percent Solids:	n/a
Project:	BMSMC, Building 5 Area, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	3W26095.D	240	02/07/12	YXC	n/a	n/a	V3W1029
Run #2	3W26108.D	12000	02/08/12	YXC	n/a	n/a	V3W1030

Run #	Initial Volume
Run #1	40.0 ml
Run #2	100 ml

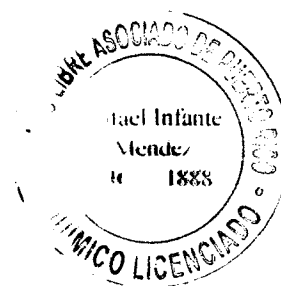
Purgeable Aromatics

CAS No.	MW	Compound	Result	RL	MDL	Units	Q	Result	RL	Units
71-43-2	78.11	Benzene	ND	480	110	ppbv		ND	1500	ug/m3
108-88-3	92.14	Toluene	32500	480	96	ppbv		122000	1800	ug/m3
100-41-4	106.2	Ethylbenzene	312000 ^a	9600	1500	ppbv		1360000 ^a	42000	ug/m3
1330-20-7	106.2	Xylenes (total)	922000 ^a	9600	1500	ppbv		4010000 ^a	42000	ug/m3
	106.2	m,p-Xylene	891000 ^a	9600	1500	ppbv		3870000 ^a	42000	ug/m3
95-47-6	106.2	o-Xylene	31000	480	75	ppbv		135000	2100	ug/m3
67-64-1	58.08	Acetone	112000 ^a	9600	1700	ppbv		266000 ^a	23000	ug/m3
67-63-0	60.1	Isopropyl Alcohol	58100	480	140	ppbv		143000	1200	ug/m3
108-10-1	100.2	Methyl Isobutyl Ketone	424000 ^a	9600	1700	ppbv		1740000 ^a	39000	ug/m3

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	4-Bromofluorobenzene	47% ^b	98%	65-128%

(a) Result is from Run# 2

(b) Outside control limits due to matrix interference.



ND = Not detected MDL - Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

Accutest LabLink@657856 13:40 15-Feb-2012

Report of Analysis

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Client Sample ID:	A-1R3A	Date Sampled:	02/02/12
Lab Sample ID:	JA98691-2	Date Received:	02/04/12
Matrix:	AIR - Soil Vapor Comp. Summa ID: A535	Percent Solids:	n/a
Method:	EPA TO-3		
Project:	BMSMC, Building 5 Area, PR		

Run #	File ID	DF	Analyzed	By	Prep Date	Prep Batch	Analytical Batch
Run #1	QR93715.D	1.2	02/07/12	TCH	n/a	n/a	GQR4341
Run #2							

Run #	Initial Volume
Run #1	0.50 ml
Run #2	

CAS No.	MW	Compound	Result	RL	MDL	Units	Q	Result	RL	Units
74-82-8	16	Methane	1200	6.0	0.89	ppmv		785	3.9	mg/m3

CAS No.	Surrogate Recoveries	Run# 1	Run# 2	Limits
460-00-4	4-Bromofluorobenzene	96%		81-115%
460-00-4	4-Bromofluorobenzene	104%		81-115%



ND = Not detected MDL - Method Detection Limit
RL = Reporting Limit
E = Indicates value exceeds calibration range

J = Indicates an estimated value
B = Indicates analyte found in associated method blank
N = Indicates presumptive evidence of a compound

AIR

ACCUTEST.
LABORATORIES

CHAIN OF CUSTODY

Air Sampling Field Data Sheet

2235 US Highway 130, Dayton, NJ 08810
Tel: 732.329.0200 Fax: 732.329.3499

FED-EX Tracking #
877046770938
Lab Quote #

Bottle Order Control # 1M-1/25/2012-6	Lab Job # JA98691
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JA98691: Chain of Custody

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DATA REVIEW WORKSHEETS

Project Number: JA98691
Date: 02/02/2012

REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from "Compendium Method TO-3. METHOD FOR THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN AMBIENT AIR USING CRYOGENIC PRECONCENTRATION TECHNIQUES AND GAS CHROMATOGRAPHY WITH FLAME IONIZATION AND ELECTRON CAPTURE DETECTION, Revision 1.1, April, 1984". The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: JA98691 Sample matrix: Air
No. of Samples: 2

Trip blank No.: -
Field blank No.: -
Equipment blank No.: -
Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input type="checkbox"/> N/A GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input type="checkbox"/> N/A Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input type="checkbox"/> N/A Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Methane_VOC's_by_method_TO-3

Definition of Qualifiers:

J- Estimated results
U- Compound not detected
R- Rejected data
UJ- Estimated nondetect

Reviewer: Rafael Infante
Date: 02/18/2012

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time				

Criteria

Aqueous samples – 14 days from sample collection for preserved samples ($\text{pH} \leq 2$, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: $4 \pm 2^{\circ}\text{C}$): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solid of soil samples is $< 10\%$, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ($> 10^{\circ}\text{C}$), estimate positive results (J) and nondetects (UJ).

DATA REVIEW WORKSHEETS

All criteria were met N/A
Criteria were not met see below _____

GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

N/A The BFB performance results were reviewed and found to be within the specified criteria.

N/A BFB tuning was performed for every 24 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List the samples affected:

If mass calibration is in error, all associated data are rejected.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 02/06/12
 Dates of continuing calibration: 02/07/12
 Instrument ID numbers: GCQR
 Matrix/Level: Air/medium

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.
 All %RSD must be $\leq 15\%$ regardless of method requirements for CCC.
 All %Ds must be $\leq 30\%$ regardless of method requirements for CCC.
 Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05 , estimate positive results (J) and reject nondetects (R), regardless of method requirements.
 If any compound has a %RSD $> 15\%$, estimate positive results (J) and use professional judgment to qualify nondetects.
 If any compound has a %RSD $> 90\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has a % D $> 30\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has a % D $> 30\%$, estimate positive results (J) and nondetects (UJ).
 If any compound has a % D $> 90\%$, estimate positive results (J) and reject nondetects (R).
 If any compound has $r > 0.995$, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
All_method_blank_meeth_method_specific_criteria				

Field/Equipment/Trip blank

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is $<$ sample quantitation limit (SQL) and \leq AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and $>$ AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below X

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
4-BFB		

QC Limits* (Air)

_____ LL to UL _____ 81 to 115 _____ to _____ to _____ to _____

4-BFB = 4-Bromofluorobenzene

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

DATA REVIEW WORKSHEETS

All criteria were met ____
 Criteria were not met ____
 and/or see below __N/A__

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: _____ Matrix/Level: _____

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
____MS/MSD_are_not_required_as_part_of_Method_TO-15;_blank_spike_used_to_assess_accuracy____					

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

- If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).
- If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).
- If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

DATA REVIEW WORKSHEETS

A separate worksheet should be used for each MS/MSD pair.

All criteria were met _____
Criteria were not met _____
and/or see below __N/A__

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: _____ Matrix/Level/Unit: _____

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
----------	-----------------	----------	-----------	-------	--------

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There are no margins or other markings on the paper.

Actions:

- * If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).
* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met _____
 and/or see below _____

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
Recoveries within laboratory control limits _____			

* QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

* If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below X

IX. FIELD DUPLICATE PRECISION

Sample IDs: JA98412-1

Matrix: Air

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD \pm 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were met N/A
Criteria were not met
and/or see below _____

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

* Area of +40% or -40% of the IS area in the associated calibration standard.
* Retention time (RT) within ± 0.06 seconds of the IS area in the associated calibration standard.

[illegible]

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

JA98691-1

Methane RF = 1,111

$$[] = (2612832)/(1.111 \times 10^3)$$

$$= 2352 \text{ ppmv OK}$$

Concentration over calibration range, laboratory qualified result as E; sample result qualified as estimated.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

XII. QUANTITATION LIMITS

A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASON FOR DILUTION
JA98641-2	1.2 x	METHANE OUTSIDE CONCENTRATIO RANGE

B. Percent Solids

List samples which have ≤ 50 % solids

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)

DATA REVIEW WORKSHEETS

Project Number: JA98691
Date: 02/02/2012

REVIEW OF VOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: QC criteria from "Compendium Method TO-15. Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999". The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest data package received has been reviewed and the quality control and performance data summarized. The data review for VOCs included:

Lab. Project/SDG No.: JA98691 Sample matrix: Air
No. of Samples: 2

Trip blank No.: -
Field blank No.: -
Equipment blank No.: -
Field duplicate No.: -

<input checked="" type="checkbox"/> Data Completeness	<input checked="" type="checkbox"/> Laboratory Control Spikes
<input checked="" type="checkbox"/> Holding Times	<input checked="" type="checkbox"/> Field Duplicates
<input checked="" type="checkbox"/> GC/MS Tuning	<input checked="" type="checkbox"/> Calibrations
<input checked="" type="checkbox"/> Internal Standard Performance	<input checked="" type="checkbox"/> Compound Identifications
<input checked="" type="checkbox"/> Blanks	<input checked="" type="checkbox"/> Compound Quantitation
<input checked="" type="checkbox"/> Surrogate Recoveries	<input checked="" type="checkbox"/> Quantitation Limits
<input type="checkbox"/> N/A Matrix Spike/Matrix Spike Duplicate	

Overall Comments: Selected VOC's by method TO-15

Definition of Qualifiers:

J- Estimated results
U- Compound not detected
R- Rejected data
UJ- Estimated nondetect

Reviewer: Rafael Infante
Date: 02/18/2012

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There are no margins, text, or other markings on the paper.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pH	ACTION
All samples analyzed within the recommended method holding time				

Criteria

Aqueous samples – 14 days from sample collection for preserved samples ($\text{pH} \leq 2$, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C , no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: $4 \pm 2^{\circ}\text{C}$): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimate positive results (J) and nondetects (UJ).

If the % solid of soil samples is $< 10\%$, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted ($> 10^{\circ}\text{C}$), estimate positive results (J) and nondetects (UJ).

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met see below

GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

 X The BFB performance results were reviewed and found to be within the specified criteria.

 X BFB tuning was performed for every 24 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

List the samples affected:

If mass calibration is in error, all associated data are rejected.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 01/16/12 01/23/12
 Dates of continuing calibration: 01/23/12 02/06/12 02/07/12 02/08/12 02/13/12
 Instrument ID numbers: GCMS2W GCMS3W
 Matrix/Level: Air/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be $\leq 15\%$ regardless of method requirements for CCC.

All %Ds must be $\leq 30\%$ regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05 , estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD $> 15\%$, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD $> 90\%$, estimate positive results (J) and reject nondetects (R).

If any compound has a % D $> 30\%$, estimate positive results (J) and reject nondetects (R).

If any compound has a % D $> 30\%$, estimate positive results (J) and nondetects (UJ).

If any compound has a % D $> 90\%$, estimate positive results (J) and reject nondetects (R).

If any compound has r > 0.995 , estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/MATRIX	COMPOUND	CONCENTRATION UNITS
			All_method_blank_meeth_method_specific_criteria	
			Summa_canisters_met_cleaning_certification_criteria	

Field/Equipment/Trip blank

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

V B. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \leq AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is > SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below X

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
	4-BFB	
JA98691-1	44.0%	No action taken; the
JA98691-2	47.0%	method does not
		have a surrogate
		recovery criteria.

QC Limits* (Air)

LL to UL 65 to 128 to to to

4-BFB = 4-Bromofluorobenzene

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 80 – 120 % for aqueous and 70 – 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

DATA REVIEW WORKSHEETS

All criteria were met ____
 Criteria were not met ____
 and/or see below __N/A__

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: _____ Matrix/Level: _____

MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
____MS/MSD_are_not_required_as_part_of_Method_TO-15;_blank_spike_used_to_assess_accuracy____					

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (JJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

DATA REVIEW WORKSHEETS

All criteria were met _____
Criteria were not met _____
and/or see below N/A

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD – Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID: _____ Matrix/Level/Unit: _____

COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION
----------	-----------------	----------	-----------	-------	--------

This image shows a single sheet of white paper with horizontal ruling lines. The lines are evenly spaced and run across the width of the page. There are no margins or other markings on the paper.

Actions:

- * If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).
* If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below _____

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD?
 Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

LCS ID	COMPOUND	% R	QC LIMIT
Recoveries within laboratory control limits			

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? Yes or No.

If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below X

IX. FIELD DUPLICATE PRECISION

Sample IDs: F89892-1/-DUP

Matrix: Air

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD \pm 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
F89892-1/-DUP					No action taken concentration < 5 x SQL
Toluene	0.80	0.65 I	0.49 J	40	

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below _____

IX. FIELD DUPLICATE PRECISION

Sample IDs: JA98624-2/-2DUP

Matrix: Air

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD \pm 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +40% or -40% of the IS area in the associated calibration standard.
- * Retention time (RT) within ± 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
------	-----------	--------	---------	------------------	--------

Internal standard area and retention times within laboratory control limits for both samples and calibration standards

Actions:

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%		IS AREA > + 40%
Positive results	J		J
Nondetected results	R		ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

JA98641-1

Benzene RF = 0.965

$$[] = (4957)(10)/(362319)(0.965)$$

$$= 0.14 \text{ ppbv OK}$$

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

XII. QUANTITATION LIMITS

A. Dilution performed

[illegible]

B. Percent Solids

List samples which have $\leq 50\%$ solids

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)